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L25: Entry 1 of 1

File: DWPI

Mar 13, 1997

DERWENT-ACC-NO: 1997-166726

DERWENT-WEEK: 199829

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TITLE: Uniform free radical polymerisation of water-insol. and opt. soluble monomers in water - using complex or mixt. of ethylenically unsatd. monomer and cyclodextrin or deriv., useful e.g. as thickener, coating, dispersant, emulsifier or in leather mfr.

INVENTOR: BORN, M; JEROMIN, J ; KISTENMACHER, A ; NOLL, O ; RITTER, H ; SCHORNICK, G

PATENT-ASSIGNEE: BASF AG (BADI)

PRIORITY-DATA: 1995DE-1033269 (September 8, 1995)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
DE 19533269 A1	March 13, 1997		007	C08F002/10
EP 848722 A1	June 24, 1998	G	000	C08F002/16
WO 9709354 A1	March 13, 1997	G	020	C08F002/16

DESIGNATED-STATES: DE FR GB IT CA JP US AT BE CH DE DK ES FI FR GB GR IE IT LU MC NL PT SE

CITED-DOCUMENTS:1.Jnl.Ref; EP 334515 ; EP 710675 ; GB 932389 ; JP 58168603

APPLICATION-DATA:

PUB-NO	APPL-DATE	APPL-NO	DESCRIPTOR
DE 19533269A1	September 8, 1995	1995DE-1033269	
EP 848722A1	September 4, 1996	1996EP-0931021	
EP 848722A1	September 4, 1996	1996WO-EP03890	
EP 848722A1		WO 9709354	Based on
WO 9709354A1	September 4, 1996	1996WO-EP03890	

INT-CL (IPC): C08 B 37/16; C08 F 2/10; C08 F 2/16

ABSTRACTED-PUB-NO: DE 19533269A

BASIC-ABSTRACT:

In free radical polymerisation of water-insol. monomers (I) and opt. water-soluble monomers (II) in a diluent, (i) water is used as diluent; and (ii) (I) are used in the form of complexes of (a) cyclodextrins. (IIIA) or cpds. (IIIB) with cyclodextrin structure and (b) ethylenically unsatd. monomers (I), which are insol. in water or have a solubility in water not more than 20 g/l at 20 deg. C, in an (a):b) molar ratio of 1:2 to 10:1; or (iii) the monomers are polymerised in the presence of not more than 5 mole (IIIA) and/or (IIIB)/mole (I).

USE - The hydrophobically modified polymers are useful as thickeners, e.g. in cosmetic cremes or lotions, in lacquers, for sizing paper, as coating compsns., in

adhesives and washing agents, as dispersants for pigments, as tanning, re-tanning, dubbing or waterproofing agents for leather mfr. and as polymeric emulsifiers for stabilising fine dispersions of apolar substances in polar phases. Crosslinked polyacrylic acids obtd. by this method are useful as super-absorbers or thickeners for aq. systems.

ADVANTAGE - Uniform homo- and copolymers are obtd.

ABSTRACTED-PUB-NO: DE 19533269A

EQUIVALENT-ABSTRACTS:

CHOSEN-DRAWING: Dwg.0/0

DERWENT-CLASS: A18 D13 D18 D21 D25 F09 G02 G03

CPI-CODES: A02-A03; A04-A; D07-B; D08-B; F05-A06B; F05-A06C; G02-A05; G02-A05C; G03-B02;

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L34: Entry 2 of 12

File: USPT

Dec 10, 2002

DOCUMENT-IDENTIFIER: US 6492450 B1

TITLE: Use of polymers in gypsum wallboard

Brief Summary Text (11):

The polymer used in this invention may be prepared by a single stage or multi-stage process. The process for making the polymer may be solution polymerization or emulsion polymerization utilizing methyl-.beta.-cyclodextrin ("CD"). See U.S. Pat. No. 5,521,266 for a detailed description of the emulsion polymerization process. The emulsion polymerization process utilizing CD is preferred.

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L34: Entry 3 of 12

File: USPT

Apr 17, 2001

DOCUMENT-IDENTIFIER: US 6218464 B1

TITLE: Preparation of fluorinated polymers

Brief Summary Text (5):

U.S. Pat. No. 5,521,266 (Lau) discloses the emulsion polymerization of hydrophobic hydrocarbon monomers using cyclodextrin. The presence of cyclodextrin facilitates the transport of hydrophobic long alkyl chain monomers through the aqueous phase so that they can be homo- or copolymerized in a conventional emulsion polymerization process. There is no disclosure of the applicability of this method of emulsion polymerization to perfluorinated monomers.

Brief Summary Text (6):

The incompatability of fluorocarbons with hydrocarbons or water is well known, as evidenced by the use of fluorocarbon coatings for non-stick cooking surfaces. The cavity of cyclodextrin is a sugar ring having hydroxylated hydrocarbons. That cyclodextrins can be used to transport fluorinated monomers through the aqueous phase in emulsion polymerizations is unexpected and was not recognized in U.S. Pat. No. 5,521,266.

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L34: Entry 6 of 12

File: USPT

Jul 7, 1998

DOCUMENT-IDENTIFIER: US 5777003 A

TITLE: Redispersible polymer powder composition comprising cyclodextrins or cyclodextrin derivatives

Brief Summary Text (45):

a) emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or cyclodextrin derivatives of the general formula (I) and if appropriate emulsifiers and in the absence of protective colloids and

Brief Summary Text (47):

The aqueous polymer dispersions are prepared by the process of aqueous emulsion polymerization in the presence of 0.3 to 10.0% by weight, based on the total weight of the monomers, of cyclodextrin or cyclodextrin derivatives of the formula (I). The polymerization temperature is as a rule 35.degree.-95.degree. C., preferably 40.degree.-80.degree. C. The polymerization medium employed is water, or if appropriate a mixture of water and solvents which are readily miscible with water, such as acetone or ethanol. The polymerization is preferably carried out in water. The polymerization can be carried out by the batch process, in which all the components are initially introduced into the reactor, and by the metering process, in which individual or several components are fed in during the polymerization. Mixed types with an initial mixture and metering are preferred. The meterings can be carried out separately (with respect to space and time), or the components to be metered can be metered entirely or in part in pre-emulsified form. The cyclodextrins or cyclodextrin derivatives can be initially introduced into the reactor or metered in, or distributed between the initial mixture and metering. Preferably, the cyclodextrins or cyclodextrin derivatives are initially introduced into the reactor. The meterings can be carried out at a constant rate and at varying rates. The same provisos as for the use of the other reaction components apply to the use of the initiator system.

Brief Summary Text (53):

The invention furthermore relates to aqueous polymer dispersions having a solids content of 30 to 75% by weight which comprise homo- or copolymers of ethylenically unsaturated monomers and cyclodextrin or cyclodextrin derivatives of the general formula (I) and are obtainable by emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or cyclodextrin derivatives of the general formula (I), and if appropriate an emulsifier, and in the absence of protective colloids.

CLAIMS:

7. A process for the preparation of a redispersible polymer powder composition which comprises a homo- or copolymer of ethylenically unsaturated monomers and cyclodextrin or a cyclodextrin derivative of the general formula (I) as claimed in claim 1

a) emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or a cyclodextrin derivative of the general formula (I) and if appropriate emulsifier and in the absence of protective colloids and

b) drying of the aqueous polymer dispersion obtained by this procedure, if appropriate with the addition of antiblocking agents and/or further additives.

8. An aqueous polymer dispersion having a solids content of 30 to 75% by weight which comprises a homo- or copolymer of ethylenically unsaturated monomers and cyclodextrin or a cyclodextrin derivative of the general formula (I) as claimed in claim 1, obtainable by emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or a cyclodextrin derivative of the general formula (I) and if appropriate emulsifier and in the absence of a protective colloids.

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L35: Entry 5 of 10

File: USPT

Jul 7, 1998

DOCUMENT-IDENTIFIER: US 5777003 A

TITLE: Redispersible polymer powder composition comprising cyclodextrins or cyclodextrin derivitaves

Brief Summary Text (45):

a) emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or cyclodextrin derivatives of the general formula (I) and if appropriate emulsifiers and in the absence of protective colloids and

Brief Summary Text (47):

The aqueous polymer dispersions are prepared by the process of aqueous emulsion polymerization in the presence of 0.3 to 10.0% by weight, based on the total weight of the monomers, of cyclodextrin or cyclodextrin derivatives of the formula (I). The polymerization temperature is as a rule 35.degree.-95.degree. C.; preferably 40.degree.-80.degree. C. The polymerization medium employed is water, or if appropriate a mixture of water and solvents which are readily miscible with water, such as acetone or ethanol. The polymerization is preferably carried out in water. The polymerization can be carried out by the batch process, in which all the components are initially introduced into the reactor, and by the metering process, in which individual or several components are fed in during the polymerization. Mixed types with an initial mixture and metering are preferred. The meterings can be carried out separately (with respect to space and time), or the components to be metered can be metered entirely or in part in pre-emulsified form. The cyclodextrins or cyclodextrin derivatives can be initially introduced into the reactor or metered in, or distributed between the initial mixture and metering. Preferably, the cyclodextrins or cyclodextrin derivatives are initially introduced into the reactor. The meterings can be carried out at a constant rate and at varying rates. The same provisos as for the use of the other reaction components apply to the use of the initiator system.

Brief Summary Text (52):

The aqueous dispersions obtainable by the process according to the invention have a solids content of 30 to 75% by weight, preferably 40 to 65% by weight.

Brief Summary Text (53):

The invention furthermore relates to aqueous polymer dispersions having a solids content of 30 to 75% by weight which comprise homo- or copolymers of ethylenically unsaturated monomers and cyclodextrin or cyclodextrin derivatives of the general formula (I) and are obtainable by emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or cyclodextrin derivatives of the general formula (I), and if appropriate an emulsifier, and in the absence of protective colloids.

Brief Summary Text (56):

For drying, the dispersions are brought to a solids content of 10 to 75% by weight, preferably 30 to 65% by weight. The solids content depends on the drying process chosen and on the nature and amount of further additives which are added during drying. For the preferred spray drying, a viscosity of the overall system of up to 1000 mPa.s has proved appropriate.

Detailed Description Text (4):

101 kg of completely desalinated water, 3.0 kg of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate and 1.65 kg of 10% strength acetic acid were initially introduced into an autoclave. The pH of the initial mixture was brought to 4.0 with 10% strength sodium hydroxide solution. 22.5 kg of vinyl acetate were emulsified in, while stirring, and the mixture was heated up to 75.degree. C. When temperature equilibrium had been reached, ethylene was forced in up to 20 bar. Metering of solutions of 630 g of potassium peroxodisulfate in 20.4 kg of water and 316 g of hydroxymethanesulfinic acid in 20.8 kg of water was then started simultaneously. After the start of the reaction, 203 kg of vinyl acetate were allowed to run in. A solution of 113 kg of water, 20 kg of 30% strength sodium lauryl polyglycol sulfate and 2.25 kg of 50% strength sodium acrylamido-2-methylpropanesulfonate was also metered in. When the monomer and emulsifier feed had ended, also no further ethylene was fed in, and the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled and the autoclave was let down. To bring the polymerization to completion, 1.94 kg of 10% strength aqueous tert-butyl hydroperoxide solution and 1.94 kg of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (5):

An ethylene/vinyl acetate copolymer with an ethylene content of 15.0% by weight, a vinyl acetate content of 84.6% by weight and 0.4% by weight of AMPS resulted. The dispersion had no coarse fraction, and had a solids content of 49.8% by weight, a pH of 4.0 and an average particle size of 310 nm.

Detailed Description Text (7):

349 g of completely desalinated water, 5.12 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate and 6.00 g of 10% strength acetic acid were initially introduced into a glass reactor. The pH of the initial mixture was brought to 4.0 with 10% strength sodium hydroxide solution. 41.0 g each of styrene and butyl acrylate were emulsified in, while stirring, and the mixture was heated up to 50.degree. C. 320 g of water, 55.6 g of 30% strength sodium lauryl polyglycol sulfate, 81.9 g of 30% strength acrylamide, 369 g of styrene and 369 g of butyl acrylate were pre-emulsified in a metering container. The pH of the pre-emulsion was brought to 4.0 with 10% strength acetic acid. Metering in of solutions of 12.9 g of 40% strength tert-butyl hydroperoxide in 159 g of water and 8.82 g of hydroxy-5-methanesulfinic acid in 163 g of water was then started simultaneously, while stirring. After the start of the reaction, the pre-emulsion was metered in uniformly. When the pre-emulsion feed had ended, the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled. To bring the polymerization to completion, 5.3 ml of 10% strength aqueous tert-butyl hydroperoxide solution and 4.3 ml of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (8):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion had no coarse fraction and had a solids content of 49.0% by weight, a pH of 3.5 and an average particle size of 210 nm.

Detailed Description Text (10):

17.0 kg of completely desalinated water, 923 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate, 270 g of 10% strength acetic acid and 1.85 kg of a-cyclodextrin were initially introduced into a polymerization tank. The pH of the initial mixture was brought to 4.1 with 10% strength sodium hydroxide solution. 1.85 kg each of styrene and butyl acrylate were emulsified in, while stirring, and the mixture was heated up to 50.degree. C. 14.4 kg of water, 2.46 kg of 30% strength sodium lauryl polyglycol sulfate, 3.69 kg of 30% strength acrylamide, 16.6 kg of styrene and 16.6 kg of butyl acrylate were pre-emulsified in a metering container. The pH of the pre-emulsion was brought to 4.1 with 10% strength acetic acid. Metering of solutions of 581 g of 40% strength tert-butyl hydroperoxide in 7.17 kg of water and 399 g of hydroxymethanesulfinic acid in 7.39 kg of water was then started simultaneously, while stirring. After the start of these meterings, the pre-emulsion was metered in. When the pre-emulsion feed had ended, the initiator

system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled. To bring the polymerization to completion, 240 ml of 10% strength aqueous tert-butyl hydroperoxide solution and 240 g of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (11):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 44.6% by weight, a pH of 3.7 and an average particle size of 350 nm.

Detailed Description Text (14):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 48.2% by weight, a pH of 4.4 and an average particle size of 300 nm.

Detailed Description Text (17):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 46.7% by weight, a pH of 3.7 and an average particle size of 260 nm.

Detailed Description Text (20):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 46.2% by weight, a pH of 3.3 and an average particle size of 290 nm.

Detailed Description Text (23):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 45.7% by weight, a pH of 3.4 and an average particle size of 300 nm.

Detailed Description Text (26):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 46.9% by weight, a pH of 4.0 and an average particle size of 1700 nm.

Detailed Description Text (29):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 44.8% by weight, a pH of 4.1 and an average particle size of 2000 nm.

Detailed Description Text (32):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 43.2% by weight, a pH of 4.2 and an average particle size of 300 nm.

Detailed Description Text (34):

2580 g of completely desalinated water, 127 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate, 46.5 ml of 10% strength acetic acid and 317 g of acetyl-.beta.-cyclodextrin (degree of substitution 1.0) (Ac-.beta.-CD, DS=1.0) were initially introduced into a pressure reactor. The pH of the initial mixture was 4.0. 635 g of vinyl acetate were emulsified in, while stirring, and the mixture was heated up to 75.degree. C. When temperature equilibrium had been reached, up to 25 bar of ethylene were forced in. Metering of solutions of 47.7 g of potassium peroxodisulfate in 1540 g of water and 23.8 g of hydroxymethanesulfinic acid in 1560 g of water were then started simultaneously. After the start of the reaction, 5710 g of vinyl acetate and a solution of 265 g of 30% strength sodium lauryl polyglycol sulfate and 79.4 g of 50% strength sodium

acrylamido-2-methylpropanesulfonate in 3960 g of water were metered in. When the monomer and emulsifier feed had ended, no further ethylene was fed in and the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled and the autoclave was let down. To bring the polymerization to completion, 55 g of 10% strength aqueous tert-butyl hydroperoxide solution and 55 g of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (35):

An ethylene/vinyl acetate copolymer with an ethylene content of 15% by weight, a vinyl acetate content of 84.6% by weight and 0.4% by weight of AMPS resulted. The dispersion had no coarse fraction and had a solids content of 50.5% by weight, a pH of 3.2 and an average particle size of 570 nm.

Detailed Description Text (38):

An ethylene/vinyl acetate copolymer with an ethylene content of 12.0% by weight, a vinyl acetate content of 87.6% by weight and 0.4% by weight of AMPS resulted. The dispersion had no coarse fraction and had a solids content of 55% by weight, a pH of 3.0 and an average particle size of 260 nm.

Detailed Description Text (41):

An ethylene/vinyl acetate copolymer with an ethylene content of 12.0% by weight, a vinyl acetate content of 87.6% by weight and 0.4% by weight of AMPS resulted. The dispersion had no coarse fraction and had a solids content of 49.6% by weight, a pH of 3.0 and an average particle size of 500 nm.

Detailed Description Text (44):

An ethylene/vinyl acetate copolymer with an ethylene content of 9.0% by weight, a vinyl acetate content of 90.5% by weight and 0.5% by weight of AMPS resulted. The dispersion had no coarse fraction and had a solids content of 52.3% by weight, a pH of 4.9 and an average particle size of 770 nm.

Detailed Description Text (47):

An ethylene/vinyl acetate copolymer with an ethylene content of 15.0% by weight, a vinyl acetate content of 84.6% by weight and 0.4% by weight of AMPS resulted. The dispersion had no coarse fraction and had a solids content of 50.9% by weight, a pH of 2.9 and an average particle size of 500 nm.

Detailed Description Text (49):

2480 g of completely desalinated water, 143 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate and 52.3 ml of 10% strength acetic acid were initially introduced into a pressure reactor. The pH of the initial mixture was brought to 4.0 with 10% strength sodium hydroxide solution. 714 g of vinyl acetate were emulsified in, while stirring, and the mixture was heated up to 75.degree. C. When temperature equilibrium had been reached, up to 25 bar of ethylene were forced in. Metering in of solutions of 53.7 g of potassium peroxodisulfate in 1730 g of water and 26.8 g of hydroxymethanesulfinic acid in 1760 g of water was then started simultaneously. After the start of the reaction, 6430 g of vinyl acetate and a solution of 714 g of 30% strength sodium lauryl polyglycol sulfate, 71.4 g of 50% strength sodium acrylamido-2-methylpropanesulfonate and 143 g of acetyl-.beta.-cyclodextrin (DS =1.0) in 3570 g of water were metered in continuously. When the monomer and emulsifier feed had ended, also no further ethylene was fed in and the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled and the autoclave was let down. To bring the polymerization to completion, 61 ml of 10% strength aqueous tert-butyl hydroperoxide solution and 61 ml of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (50):

An ethylene/vinyl acetate copolymer with an ethylene content of 9.0% by weight, a vinyl acetate content of 90.5% by weight and 0.5% by weight of AMPS resulted. The dispersion contained hardly any coarse fraction and had a solids content of 51.7% by weight, a pH of 3.3 and an average particle size of 240 nm.

Detailed Description Text (53):

An ethylene/vinyl acetate copolymer with an ethylene content of 15.0% by weight, a vinyl acetate content of 84.6% by weight and 0.4% by weight of AMPS resulted. The dispersion contained hardly any coarse fraction and had a solids content of 50.4% by weight, a pH of 3.1 and an average particle size of 310 nm.

Detailed Description Text (55):

341 g of completely desalinated water, 18.5 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate and 5.41 g of 10% strength acetic acid were initially introduced into a glass reactor. The pH of the initial mixture was brought to 4.0 with 10% strength sodium hydroxide solution. 36.9 g each of styrene and butyl acrylate were emulsified in, while stirring, and the mixture was heated up to 50.degree. C. 288 g of water, 45.2 g of 30% strength sodium lauryl polyglycol sulfate, 73.8 g of 30% strength acrylamide, 332 g of styrene, 332 g of butyl acrylate and 36.9 g of acetyl-.beta.-cyclodextrin (DS=1.0) were pre-emulsified in a metering container. The pH of the pre-emulsion was brought to 4.0 with 10% strength acetic acid. Metering in of solutions of 11.6 g of 40% strength tert-butyl hydroperoxide in 144 g of water and 7.95 g of hydroxymethanesulfinic acid in 148 g of water was then started simultaneously, while stirring. After the start of the reaction, the pre-emulsion was metered in uniformly. When the pre-emulsion feed had ended, the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled. To bring the polymerization to completion, 4.8 ml of 10% strength aqueous tert-butyl hydroperoxide solution and 4.8 g of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (56):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 48.2% by weight, a pH of 3.3 and an average particle size of 220 nm.

Detailed Description Text (59):

A styrene/butyl acrylate copolymer with a styrene content of 48.5% by weight, a butyl acrylate content of 48.5% by weight and 3.0% by weight of acrylamide resulted. The dispersion contained hardly any coarse fraction and had a solids content of 48.0% by weight, a pH of 3.4 and an average particle size of 250 nm.

Detailed Description Text (61):

2.38 kg of completely desalinated water, 129 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate, 37.9 g of 10% strength acetic acid and 259 g of hydroxypropyl-.beta.-cyclodextrin (DS=0.9) were initially introduced into a glass reactor. The pH of the initial mixture was brought to 4.0 with 10% strength sodium hydroxide solution. 228 g of styrene and 290 g of butyl acrylate were emulsified in, while stirring, and the mixture was heated up to 50.degree. C. 2.02 kg of water, 345 g of 30% strength sodium lauryl polyglycol sulfate, 517 g of 30% strength acrylamide, 2.05 kg of styrene and 2.61 kg of butyl acrylate were pre-emulsified in a metering container. The pH of the pre-emulsion was brought to 4.0 with 10% strength acetic acid. Metering in of solutions of 81.6 g of 40% strength tert-butyl hydroperoxide in 1.00 kg of water and 55.7 g of hydroxymethanesulfinic acid in 1.03 kg of water was then started simultaneously, while stirring. After the start of the reaction, the pre-emulsion was metered in uniformly. When the pre-emulsion feed had ended, the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled. To bring the polymerization to completion, 34 g of 10% strength aqueous tert-butyl hydroperoxide solution and 34 g of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (62):

A styrene/butyl acrylate copolymer with a styrene content of 42.7% by weight, a butyl acrylate content of 54.4% by weight and 2.9% by weight of acrylamide resulted. The glass transition temperature was 10.degree. C. The dispersion contained hardly any coarse fraction and had a solids content of 44.2% by weight, a pH of 3.3 and an average particle size of 280 nm.

Detailed Description Text (65):

A styrene/butyl acrylate copolymer with a styrene content of 39.8% by weight, a butyl acrylate content of 57.3% by weight and 2.9% by weight of acrylamide resulted. The glass transition temperature was 5.degree. C. The dispersion contained hardly any coarse fraction and had a solids content of 45.2% by weight, a pH of 3.2 and an average particle size of 390 nm.

Detailed Description Text (68):

A styrene/ethylhexyl acrylate copolymer with a styrene content of 41.7% by weight, a ethylhexyl acrylate content of 55.3% by weight and 3.0% by weight of acrylamide resulted. The glass transition temperature was -5.degree. C. The dispersion contained hardly any coarse fraction and had a solids content of 44.9% by weight, a pH of 3.6 and an average particle size of 430 nm.

Detailed Description Text (70):

17.0 kg of completely desalinated water, 923 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate, 270 g of 10% strength acetic acid and 1.85 kg of hydroxypropyl-.beta.-cyclodextrin (DS=0.9) were initially introduced into a polymerization tank. The pH of the initial mixture was brought to 4.1 with 10% strength sodium hydroxide solution. 1.85 kg each of styrene and butyl acrylate were emulsified in, while stirring, and the mixture was heated up to 50.degree. C. 14.4 kg of water, 2.46 kg of 30% strength sodium lauryl polyglycol sulfate, 16.6 kg of styrene and 16.6 kg of butyl acrylate were pre-emulsified in a metering container. The pH of the pre-emulsion was brought to 4.1 with 10% strength acetic acid. Metering of solutions of 581 g of 40% strength tert-butyl hydroperoxide in 7.17 kg of water and 399 g of hydroxymethanesulfinic acid in 7.39 kg of water was then started simultaneously, while stirring. After the start of these meterings, the pre-emulsion was metered in uniformly. When the pre-emulsion feed had ended, the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled. To bring the polymerization to completion, 240 ml of 10% strength aqueous tert-butyl hydroperoxide solution and 240 g of 10% strength aqueous hydroxymethanesulfinic acid solution were now added. A styrene/butyl acrylate copolymer with a styrene content of 50.0% by weight, a butyl acrylate content of 50.0% by weight resulted. The dispersion contained hardly any coarse fraction and had a solids content of 45.7% by weight, a pH of 3.4 and an average particle size of 300 nm.

Detailed Description Text (72):

264 g of completely desalinated water, 15.2 g of a 20% strength aqueous solution of the sodium salt of dihexylsulfosuccinate and 5.57 ml of 10% strength acetic acid were initially introduced into a laboratory autoclave. The pH of the initial mixture was brought to 4.0 with 10% strength sodium hydroxide solution. 76 g of vinyl acetate were emulsified in, while stirring, and the mixture was heated up to 75.degree. C. When temperature equilibrium had been reached, up to 25 bar of ethylene were forced in. 380 g of water, 15.2 g of acetyl-.beta.-cyclodextrin (DS=1.0), 7.6 g of 50% strength sodium acrylamido-2-methylpropanesulfonate, 76.0 g of 30% strength sodium lauryl polyglycol sulfate and 684 g of vinyl acetate were emulsified in a pre-emulsion vessel. Metering in of solutions of 6.65 g of potassium peroxodisulfate in 215 g of water and 3.33 g of hydroxymethanesulfinic acid in 218 g of water was then started simultaneously, while stirring. After the start of the reaction, the pre-emulsion was metered in continuously. During this period, the ethylene pressure was kept at 25 bar. The ethylene feed was then closed and the initiator system was still metered in further until the solids content of the dispersion no longer rose. The mixture was then cooled and the autoclave was let down. To bring the polymerization to completion, 6.5 ml of 10% strength aqueous tert-butyl hydroperoxide solution and 6.5 ml of 10% strength aqueous hydroxymethanesulfinic acid solution were now added.

Detailed Description Text (73):

An ethylene/vinyl acetate copolymer with an ethylene content of 15.0% by weight, a vinyl acetate content of 84.6% by weight and 0.4% by weight of AMPS resulted. The dispersion contained hardly any coarse fraction and had a solids content of 53.2% by weight, a pH of 3.4 and an average particle size of 330 nm.

Detailed Description Text (76):

4000 parts by weight of the dispersion from Example 5 and 244.9 parts by weight of

phenolsulfonic acid-formaldehyde condensate as a 51.0% strength solution in water (7.0% by weight, based on the dispersion solids content) were mixed thoroughly. The mixture was sprayed through a two-component nozzle. Air precompressed to 4 bar served as the spraying component; the drops formed were dried in co-current with air heated to 95.degree. C. 10.0% by weight, based on the total weight of polymeric constituents, of a commercially available antiblocking agent (mixture of calcium magnesium carbonate and magnesium hydrosilicate) was added to the resulting powder.

EXAMPLE 24

Detailed Description Text (77):

4000 parts by weight of the dispersion from example 5 and 426.7 parts by weight of naphthalene-sulfonic acid-formaldehyde condensate as a 30.0% strength solution in water (7.0% by weight, based on the dispersion solids content) were mixed thoroughly. The mixture was sprayed through a two-component nozzle. Air precompressed to 4 bar served as the spraying component; the drops formed were dried in co-current with air heated to 80.degree. C. 10.0% by weight, based on the total weight of polymeric constituents, of a commercially available antiblocking agent (mixture of calcium magnesium carbonate and magnesium hydrosilicate) was added to the resulting powder.

Detailed Description Text (79):

4000 parts by weight of the dispersion from Example 5 and 609.3 parts by weight of naphthalene-sulfonic acid-formaldehyde condensate as a 30.0% strength solution in water (10.0% by weight, based on the dispersion solids content) were mixed thoroughly. The mixture was sprayed through a two-component nozzle. Air precompressed to 4 bar served as the spraying component; the drops formed were dried in co-current with air heated to 80.degree. C. 5.0% by weight, based on the total weight of polymeric constituents, of a commercially available antiblocking agent (mixture of calcium magnesium carbonate and magnesium hydrosilicate) was added to the resulting powder.

Detailed Description Text (81):

4000 parts by weight of the dispersion from Comparison Example 2 and 664.7 parts by weight of phenolsulfonic acid-formaldehyde condensate as a 43.6% strength solution in water (15.0% by weight, based on the dispersion solids content) were mixed thoroughly. The mixture was sprayed through a two-component nozzle. Air precompressed to 4 bar served as the spraying component; the drops formed were dried in co-current with air heated to 80.degree. C. 5% by weight, based on the total weight of polymeric constituents, of a commercially available antiblocking agent (mixture of calcium magnesium carbonate and magnesium hydrosilicate) was added to the resulting powder.

Detailed Description Paragraph Table (5):

TABLE 4

Composition of the polymer dispersions Amount of Particle Comonomers Cyclo- CD (%) by Tg FC size Example Copolymer (% by weight) dextrin weight) DS (.degree.C.) (%) pH (nm)

C.Ex. 1	VAc/E/AMPS	84.6/15.0/0.4	-- -- --	10	49.8	4.0	310	C.Ex. 2	St/BA/AA						
48.5/48.5/3.0	-- -- --	20	49.0	3.5	210	Ex. 1	St/BA/AA	48.5/48.5/3.0	.alpha.-CD	5.0					
-- 20	44.6	3.7	350	Ex. 2	St/BA/AA	48.5/48.5/3.0	.beta.-CD	5.0	-- 15	48.2	4.4	300	Ex.		
3	St/BA/AA	48.5/48.5/3.0	.gamma.-CD	5.0	-- 15	46.7	3.7	260	Ex. 4	St/BA/AA					
48.5/48.5/3.0	Ac-.beta.-CD	5.0	1.0	15	46.2	3.3	290	Ex. 5	St/BA/AA	48.5/48.5/3.0					
Hp-.beta.-CD	5.0	0.9	15	45.7	3.4	300	Ex. 6	St/BA/AA	48.5/48.5/3.0	Hp-.beta.-CD	2.0				
0.9	15	46.9	4.0	1700	Ex. 7	St/BA/AA	48.5/48.5/3.0	Me-.beta.-CD	5.0	0.6	15	44.8	4.1		
2000	Ex. 8	St/BA/AA	48.5/48.5/3.0	CMe-.beta.-CD	5.0	0.3	15	43.2	4.2	300	Ex. 9				
VAc/E/AMPS	84.6/15.0/0.4	Ac-.beta.-CD	5.0	1.0	5	50.5	3.2	570	Ex. 10	VAc/E/AMPS					
87.6/12.0/0.4	Hp-.beta.-CD	2.0	0.9	10	55.0	3.0	260	Ex. 11	VAc/E/AMPS	87.6/12.0/0.4					
Hp-.beta.-CD	5.0	0.9	10	49.6	3.0	500	Ex. 12	VAc/E/AMPS	90.5/9.0/0.5	Me-.beta.-CD	5.0				
0.6	15	52.3	4.9	770	Ex. 13	VAc/E/AMPS	84.6/15.0/0.4	Me-.beta.-CD	5.0	1.8	5	50.9	2.9		
500	Ex. 14	VAc/E/AMPS	90.5/9.0/0.5	Ac-.beta.-CD	2.0	1.0	15	51.7	3.3	240	Ex. 15				
VAc/E/AMPS	84.6/15.0/0.4	Ac-.beta.-CD	5.0	1.0	5	50.4	3.1	310	Ex. 16	St/BA/AA					
48.5/48.5/3.0	Ac-.beta.-CD	5.0	1.0	15	48.2	3.3	220	Ex. 17	St/BA/AA	48.5/48.5/3.0					
Ac-.beta.-CD	2.0	1.0	15	48.0	3.4	250	Ex. 18	St/BA/AA	42.7/54.4/2.9	Hp-.beta.-CD	5.0				
0.9	10	44.2	3.3	280	Ex. 19	St/BA/AA	39.8/57.3/2.9	Hp-.beta.-CD	5.0	0.9	5	45.2	3.2		
390	Ex. 20	St/EHA/AA	41.7/55.3/3.0	Hp-.beta.-CD	5.0	0.9	-5	44.9	3.6	430	Ex. 21	St/BA			

50.0/50.0 Hp-.beta.-CD 5.0 0.9 15 45.7 3.4 300 Ex. 22 VAc/E/AMPS 84.6/15.0/0.4
Ac-.beta.-CD 2.0 1.0 5 53.2 3.4 330

CLAIMS:

7. A process for the preparation of a redispersible polymer powder composition which comprises a homo- or copolymer of ethylenically unsaturated monomers and cyclodextrin or a cyclodextrin derivative of the general formula (I) as claimed in claim 1

a) emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or a cyclodextrin derivative of the general formula (I) and if appropriate emulsifier and in the absence of protective colloids and

b) drying of the aqueous polymer dispersion obtained by this procedure, if appropriate with the addition of antiblocking agents and/or further additives.

8. An aqueous polymer dispersion having a solids content of 30 to 75% by weight which comprises a homo- or copolymer of ethylenically unsaturated monomers and cyclodextrin or a cyclodextrin derivative of the general formula (I) as claimed in claim 1, obtainable by emulsion polymerization of one or more ethylenically unsaturated monomers by means of free radical initiators in an aqueous medium in the presence of cyclodextrin or a cyclodextrin derivative of the general formula (I) and if appropriate emulsifier and in the absence of a protective colloids.

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L35: Entry 6 of 10

File: USPT

Jun 2, 1998

DOCUMENT-IDENTIFIER: US 5760129 A
 TITLE: Method for forming polymers

Brief Summary Text (11):

(3) the formation of emulsion or suspension polymer during a solution polymerization or large suspension polymer particles during an emulsion polymerization;

Brief Summary Text (12):

(4) phase separation or creaming of monomer droplets or large suspension particles; and

Brief Summary Text (77):

(3) the formation of large suspension polymer particles during an emulsion polymerization;

Brief Summary Text (78):

(4) phase separation or creaming of monomer droplets or large suspension particles; and

Detailed Description Text (17):

Emulsion Polymerization--Cyclodextrin Added in Kettle

Detailed Description Text (28):

Several techniques were used to determine whether the monomers having very low water solubility of Example 4 were actually incorporated randomly into the final polymer. First, lack of pooling of monomer having very low water solubility around the stirring vortex was evidence of good conversion of the monomer having very low water solubility into the final polymer. Second, lack of large 1-10 microns particles and monomer droplets, collected (as is or by centrifugation or from creamy layer) and characterized by optical microscopy, was evidence of good incorporation of the monomer having very low water solubility into the final polymer. Third, lack of formation of gel (collected through a 100 mesh screen) was evidence that the polymerization generally ran well. Finally, a single glass transition temperature at approximately the calculated copolymer values using literature data, as measured by differential scanning calorimetry at a heating rate of 20.degree. C./minute, was evidence of good incorporation of the monomer having very low water solubility to form a random copolymer. The results are shown in Table 5.1.

Detailed Description Paragraph Table (8):

TABLE 5.1 Large (1-10.mu.) Particles and
Emulsion Monomer Monomer Polymer Pooling Droplets Gel Tg (.degree.C.)

	1C-E	yes	yes	>20%	-13.6,	13.8	(Comparative)
(2 transitions) 2C-E	yes	yes	>20%	-30.7,	18.0	(Comparative)	(2 transitions)
1-E	no	<1%	17.5	2-E	no	<1%	-10.5
3-E	no	<1%	32.4	4-E	no	<1%	23.7
5-E	no	<1%	12.9	6-E	no	<1%	4.5
7-E	no	<1%	-52.2	8-E	no	10%	-53.3
9-E	no	<1%	19.3	10-E	no	<1%	11.3
11-E	no	<1%	48.8	12-E	no	<1%	58.0
13-E	no	<1%	-27.0	14-E	no	<1%	27.6
15-E	no	<1%	-10.4	16-E	no	<1%	41.8
17-E	no	<1%	50.5	18-E	no	<1%	11.2
19-E	no	<1%	19.0	20-E	no	<1%	44.4
21-E	no	<1%	36.2	22-E	no	<1%	-53.5
23-E	no	<2.1%	13.3	24-E	no	<1%	23.6
25-E	no	<1%	2.9	26-E	no	<1%	24.9



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gamma-Cyclodextrin [17465-86-0]

Synonyms: CD hydrate; -Cyclodextrin; CYCLOOCTAAMYLOSE; Cyclooctaamylose hydrate; gamma-Cyclodextrin; gamma-Cyclodextrin hydrate; gamma-Schardinger dextrin hydrate;

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Formula		Molecular Weight	
CAS RN	17465-86-0	Melting Point (°C)	267
ACX Number	X1027560-6	Boiling Point (°C)	
Density		Vapor Density	
Refractive Index		Vapor Pressure	
Evaporation Rate		Water Solubility	
Flash Point (°C)		EPA Code	
DOT Number		RTECS	
Comments			

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Dextrin [9004-53-9]

Synonyms: Dextrin; Dextrin, corn; Dextrin hydrate, White; Starch gum;

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Formula

Molecular Weight

CAS RN 9004-53-9

Melting Point (°C)

ACX Number X1001232-7

Boiling Point (°C)

Density

Vapor Density

Refractive Index

Vapor Pressure

Evaporation Rate

Water Solubility

Flash Point (°C)

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DOT Number

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